



Degradation of the Adhesive Properties of MD–944 Diode Tape by Simulated Low Earth Orbit Environmental Factors

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LIST OF ACRONYMS AND SYMBOLS

AO	atomic oxygen
AOBF	Atomic Oxygen Beam Facility
AODTS	Atomic Oxygen Drift Tube System
C	carbon
ESCA	Electron Spectroscopy for Chemical Analysis
FTIR	Fourier Transform Infrared
IPA	isopropyl alcohol
IR	ionizing radiation
ISS	International Space Station
NUV	near ultraviolet
O	oxygen
P4	port side number 4
P6	port side number 6
PSA	pressure sensitive adhesive
TM	Technical Memorandum
UV	ultraviolet
VUV	vacuum ultraviolet
XPS	x-ray photoelectron spectroscopy

TECHNICAL MEMORANDUM

DEGRADATION OF THE ADHESIVE PROPERTIES OF MD-944 DIODE TAPE BY SIMULATED LOW EARTH ORBIT ENVIRONMENTAL FACTORS

1. INTRODUCTION

On the International Space Station (ISS), the port side No. 6 (P6) solar array was deployed on top of the Z1 truss during the STS-97 mission in November 2000. Later in the ISS assembly process, the P6 array will be retracted and moved. The current operational plan is to store the P6 solar array for ≈ 6 mo after the port side No. 4 (P4) solar array is installed and becomes operational. The P6 solar array will eventually be redeployed outboard of the P4 solar array.

Originally the solar array was designed and constructed for the Space Station Freedom program, and no retraction of the array was planned after its initial deployment. During the retraction of the array, the surfaces of adjacent array panels may contact each other. In several areas on the surface of the panels, MD-944 diode tape was applied during the fabrication process (fig. 1). This tape provides thermal control by its high emittance and mechanical protection of the underlying diodes. It consists of a 0.5-mil Kapton[®] layer with a 2-mil, Dow Corning QC-7725 silicone pressure-sensitive adhesive layer. Atomic oxygen (AO), present in Space Station orbit, will chemically react with and erode the Kapton, exposing the silicone adhesive. Should the silicone retain its adhesive properties, the solar array may be mechanically damaged, or redeployment of the array may be prevented by the formation of adhesive bonds between adjacent array panels.

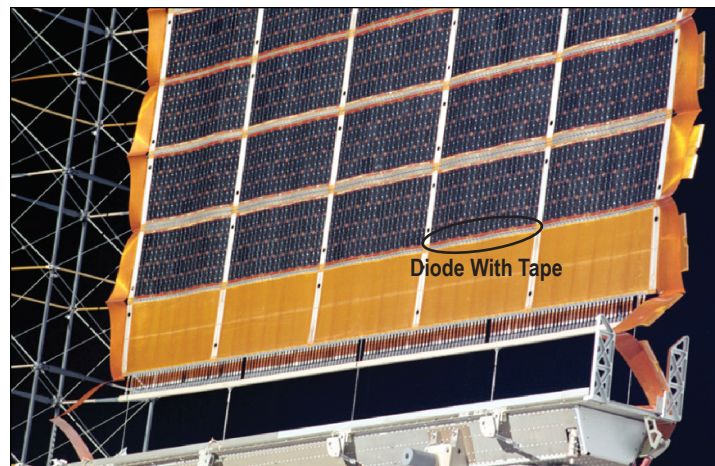


Figure 1. Diode with MD-944 tape on ISS solar array.

To evaluate the potential for the formation of adhesive bonds and the force need to overcome the bonding, samples of MD-944 diode tape have been exposed, in the Marshall Space Flight Center (MSFC) Environmental Effects Laboratory, to several of the environmental factors that the P6 solar array has experienced during its current deployment. Samples were exposed to AO of 5-eV energy, which includes a concurrent exposure to vacuum ultraviolet (VUV) radiation. AO exposure was also performed in the AO Drift Tube System, which generates thermal energy AO and no concurrent ultraviolet (UV) radiation. Tape samples have also been exposed to near ultraviolet (NUV) radiation and ionizing radiation (IR). The tape samples were exposed to each environmental factor individually and in sequence (5-eV AO, NUV, IR) in order to establish the effect of the individual and combined environment factors. After exposure to the individual and combined factors, the silicone adhesive layer of two tape samples were pressed together and placed under a “preloading” of 2.5 psi. This preloading was removed after predetermined periods of 7 days to 17 mo and the mechanical force required to break the adhesive-to-adhesive bond measured with a load frame.

During the redeployment of the P6 solar array, a force of 2.5 psi can be applied to the array by the deployment mechanism. Measured tensile forces of <2.5 psi for the separation of the silicone adhesive to silicone adhesive bond will demonstrate that a successful redeployment of the P6 solar array is possible.

2. MD-944 TAPE CONFIGURATION

As-received MD-944 tape is sandwiched between protective layers of polymer films (fig. 2). A thick Mylar® film and a film identified as SPV-367, by Lockheed-Martin, are bonded to the Kapton layer of the MD-944 tape. Both of these layers were removed before the MD-944 tape was bonded to the pressure sensitive adhesive (PSA)/substrate assembly. A Dow Corning release film covers and protects the silicone adhesive layer of the MD-944 tape. This release film was removed prior to exposing the adhesive to the various simulated environments or the sample to sample bonding of the control and blocking samples.

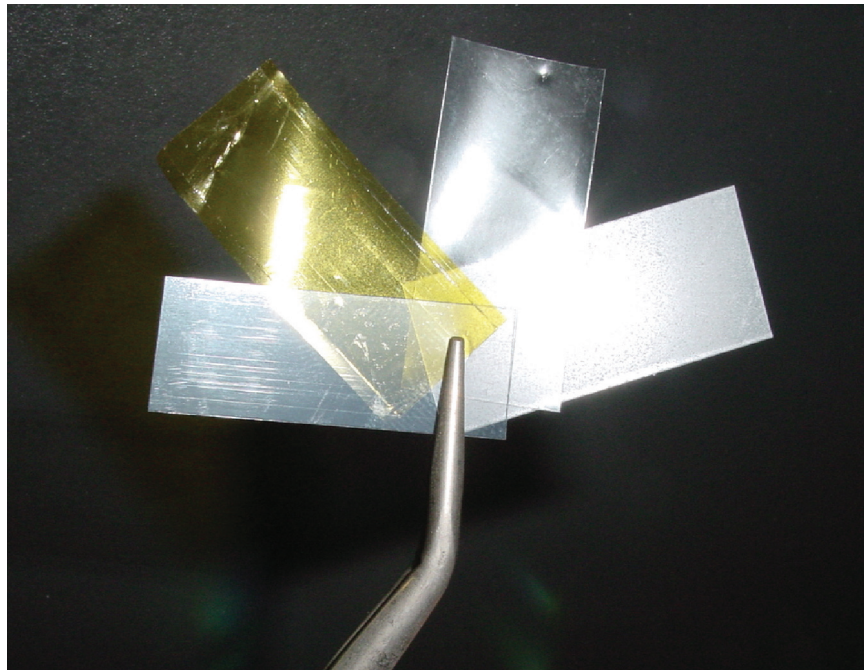


Figure 2. The four film layers of the as-received MD-944 tape. The Kapton layer of the MD-944 tape can be seen as the yellow film, which is the second film from the left.

3. SUBSTRATES

Three sizes of substrates were fabricated for this study. All of the substrates are 0.5-in-wide, 0.75-in-thick aluminum blocks. The lengths of the substrates are 1, 2, and 4 in with the 2- and 4-in-long substrates specifically fabricated for the blocking test described later in this Technical Memorandum (TM). A 0.375-in-diameter hole was drilled through the 0.75- by 1-, 2-in, or 4-in face of the substrate at the center of that face. This hole was intended for the insertion of a clevis pin for the tensile testing but was not used due to interference between the clevis and the fixture of the load frame. The face opposite the bonding surface of the substrates was drilled and tapped for a 1/4×20 bolt which was used to attach the substrate to the load frame fixture during the tensile test measurements. This hole is centered in that substrate face and intersects the 0.375-in hole in the side of the substrate. The substrates were individually numbered for tracking purposes.

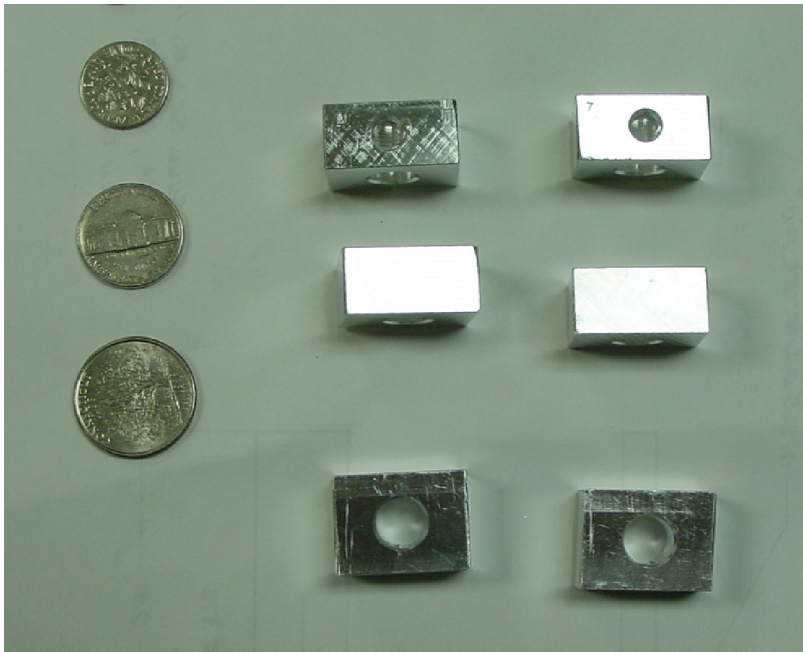


Figure 3. Several views of the aluminum substrates. The coins were included in as an indication of the substrate size.



Figure 4. Finish of the substrate bonding surface.

When the substrates were delivered for this study, they were cleaned by washing in Joy[®] detergent and water to remove oil and other contaminants from the fabrication process. This wash was followed by several soaking rinses in water before air drying the substrates. The dry substrates were then rinsed with isopropyl alcohol (IPA) and allowed to air dry before any further processing was done.

The substrates were selected randomly from the available supply of substrates when setting up the various samples required for this study. Some of the substrates were recycled, or used more than once during the study, after being solvent cleaned to remove residual pressure sensitive adhesive (PSA) from the substrate bonding surface.

The bonding face of the substrates is 0.5-in wide and 1-, 2-, or 4-in long, depending on the measurement they were intended to support. The finish or “roughness” of the bonding surface was not specified beyond a general flatness. Only a handful of the substrates were rejected due to unacceptable machining marks in that surface, and none of the substrates were further processed to produce a surface with a specified smoothness.

4. PRESSURE SENSITIVE ADHESIVE

Two 3M VHB™ PSA tapes were recommended by M. Prince (EM40) as possible candidates for this study. The tapes are 3M VHB 4920 and 3M F-9473PC. The tapes were evaluated for their ability to bond to Kapton using a layer of Kapton film to simulate that of the MD-944 tape. The Kapton/PSA bond for both tapes failed in tension above 50 psi (table 1) and the 3M VHB 4920 tape was selected and used for the remainder of the study based on these results.

Table 1. Bond strength of 3M PSA tapes to Kapton film.

Tape	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
4920	25.303	50.607
4920	34.493	68.986
4920	28.424	56.847
F9473PC	26.387	52.773
F9473PC	31.455	62.909
F9473PC	29.165	58.33

The PSA to substrate bond was also evaluated (table 2) without the layer of Kapton ®. The sample configuration was substrate/PSA/substrate and only the No. 4920 PSA was evaluated.

Table 2. Bond strength of 3M VHB 4920 to aluminum substrates.

Preload Period (days)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
7	29.282	58.564
7	28.29	56.579
7	27.968	55.935
7	34.273	68.546
7	28.856	57.712

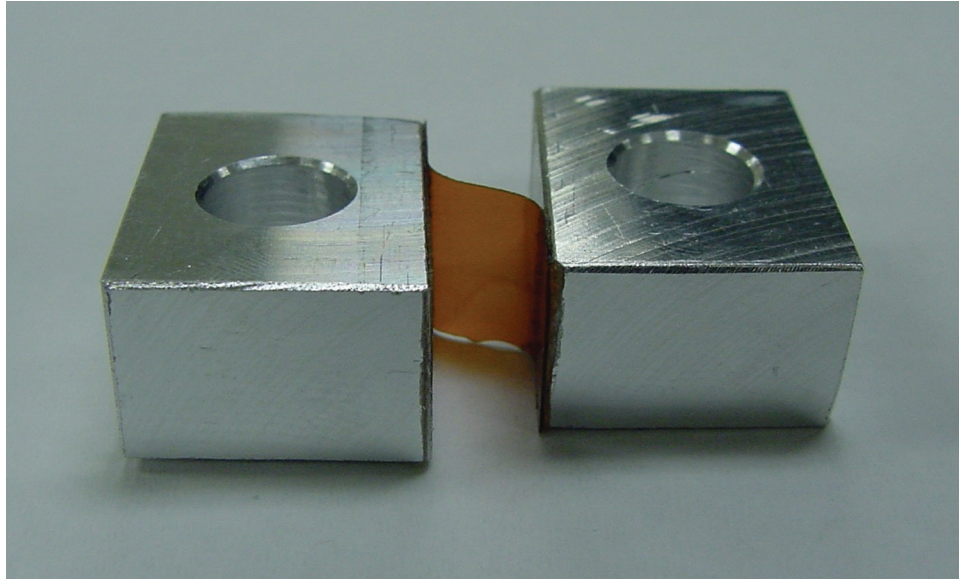


Figure 5. An after tensile test measurement sample showing the Kapton film still bonded to the substrates.

5. GENERAL SAMPLE CONFIGURATION

The general configuration of the samples consisted of a layer of the 3M VHB 4920 PSA applied to the bonding surface of the aluminum substrate. To the “top” surface of the PSA, a single layer of the MD-944 tape was applied with the Kapton bonded to the 3M VHB 4920. The silicone adhesive layer of the MD-944 tape is now the top most surface of the assembly and the bonding surface between two samples when two substrates were bonded together in the preloading and tensile test measurement configuration.

Late in the study, two new sample configurations were introduced, as a subset of samples that are identified as Kapton-up samples. Initially the silicone adhesive side of the MD-944 tape was applied directly to the 3M VHB 4920 PSA, which was already bonded to the aluminum substrate. Later the MD-944 was bonded directly to the aluminum substrate.

These samples were used to demonstrate the degradation in performance of the silicone adhesive from exposure to AO, after AO erosion of the tape’s Kapton layer. Appendix A contains the results of this additional analysis.

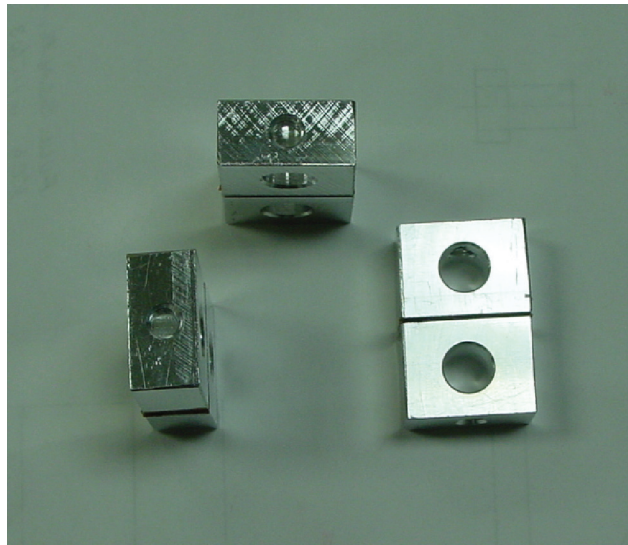


Figure 6. Several views of the substrates bonded together to form the samples prior to the preloading and tensile test measurements.

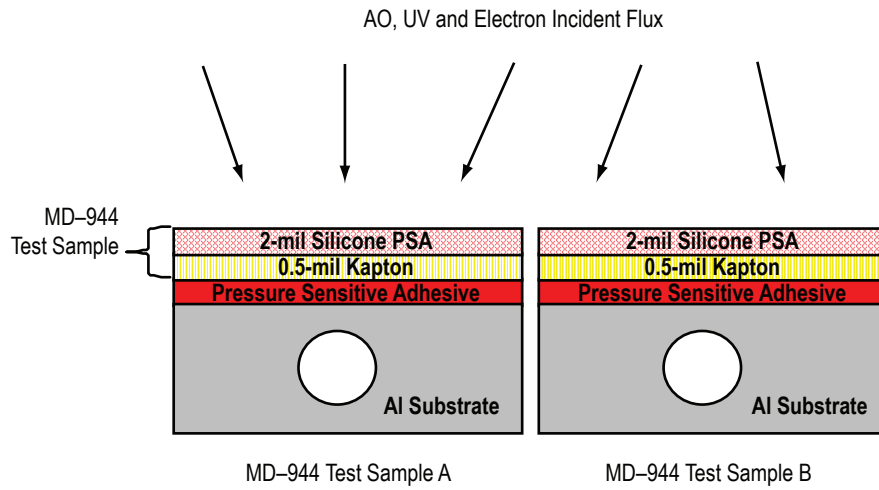


Figure 7. Substrate assembly and exposure configuration.

6. PRELOADING

Once the substrate/PSA/MD-944 substrates were assembled and exposed to the appropriate simulated environmental parameter(s), two of these substrates would be bonded together using the silicone adhesive layer of the MD-944 tape. The samples would then be “preloaded” to 2.5 psi for a period of 7 days to as long as 17 mo. The configuration of the sample being preloaded, substrate/PSA/MD-944/MD-944/PSA/substrate, was taken into account when calculating the amount of additional mass to apply to the sample to attain the 2.5-psi loading of the silicone adhesive interface, between the two MD-944 tape layers. A typical substrate mass is 14 g and the mass of the “upper” substrate was taken into account when the mass to be applied to the bond was calculated. Typically three or more samples were preloaded at the same time and an aluminum plate would be placed on top of the samples to apply the preload to the silicone adhesive bond. In this configuration the samples could be compared to the legs of a table with the aluminum plate being the table top. Like the upper substrate, the mass of the aluminum plate, typically 512 g, was accounted for when calculating the loading of the samples. In all cases, additional mass in the form of lead shot (in a metal pan) was placed on top of the aluminum plate to achieve the desired loading (figs. 8–10).

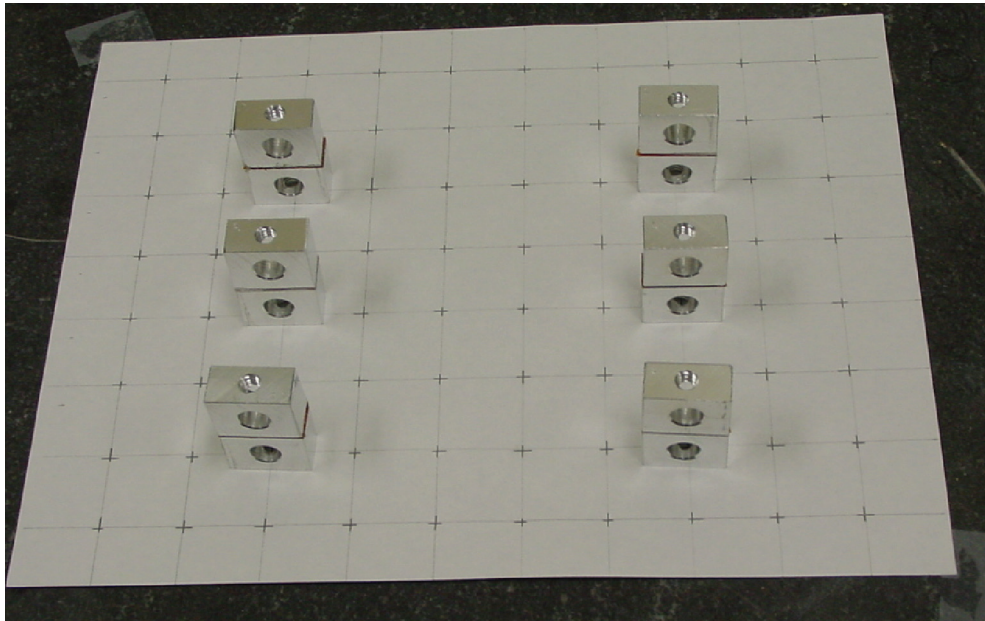


Figure 8. The assembled samples prior to application of the preload mass (the aluminum plate and lead shot).

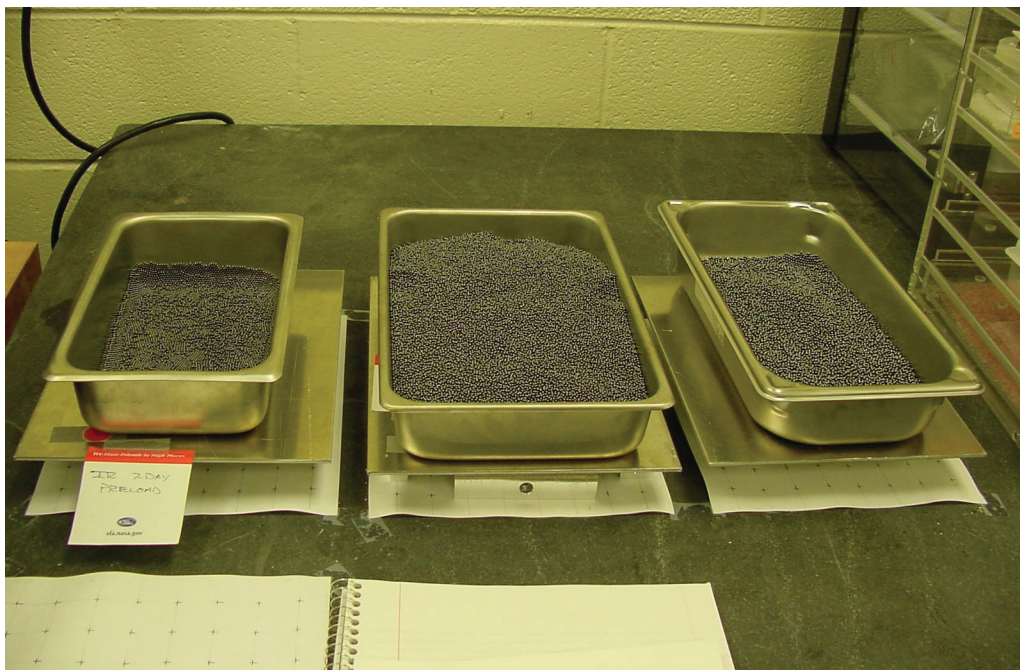


Figure 9. Preloading of the samples. Lead shot was placed in a metal pan, the amount adjusted for the weight of the plate and pan, to attain the required preload. One of the 4-in blocking samples can be seen under the center preload setup.



Figure 10. A side view of the preloading configuration.

7. TENSILE TESTING

A load frame was used to measure the force required for the bond between the layers of silicone adhesive of the MD-944 tape to fail. A target force of 2.5 psi or less, based on the amount of force the ISS solar array drive mechanism can provide for deployment, was used to evaluate the effects of the various simulated environments on the tensile strength of the silicone/silicone adhesive bond. Failure of the substrate-to-PSA bond or the PSA-to-Kapton bond was considered an erroneous result, and that tensile measurement was excluded from this study.



Figure 11. A sample mounted in the load frame prior to measuring the tensile strength of the silicone-to-silicone adhesive bond.

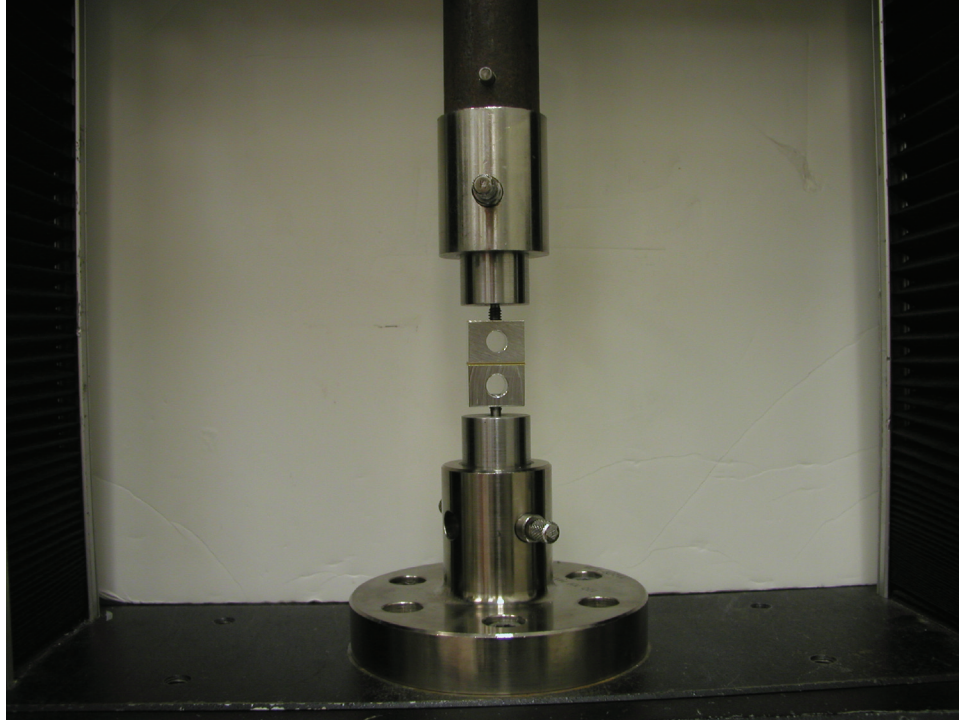


Figure 12. Another view of the samples in the load frame in the configuration used to determine the tensile strength of the bond between the substrates.

8. RECYCLED SUBSTRATES

During several phases of this study, samples were removed from the substrates, and the substrate was recycled to support another phase of the study. The MD-944 tape and the PSA were skived off the substrate and the bonding surface of the substrate lightly sanded with emery paper to expose the bare metal of the bonding surface. The entire substrate was then rinsed several times with alternating flushes of IPA and acetone and allowed to air dry before reuse.

9. CONTROL SAMPLES

All of the control samples were constructed with the 1-in-long substrates. Initial preloading of the control samples was to be for the period of 7 days, 3, 6, or 10 mo. These control samples were set up in groups of three samples for each preload period (table 3).

Table 3. Bond strength of initial set of 1-in control samples.

Preload Period	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
7 days	18.186	36.372
7 days	26.428	52.856
7 days	26.385	52.769
3 mo	12.513	25.026
3 mo	20.641	41.282
3 mo	17.34	34.68
6 mo	28.421	56.842
6 mo	33.635	67.27
6 mo	31.479	62.958
10 mo	25.176	50.353
10 mo	16.039	33.244
10 mo	27.039	54.078

10. MONTHLY CONTROL SAMPLES

After the tensile test measurements of the 7-day preload samples were completed, additional monthly control samples were added to the study. These samples (table 4) were set up in groups of four and were pulled at 1, 2, 4, and 5 mo as a supplement to the original control samples.

Table 4. Bond strength of 1-in monthly control samples.

Preload Period (mo)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
1	28.069	56.139
1	25.325	50.65
1	24.816	49.632
1	30.396	60.791
2	21.887	43.774
2	20.69	41.38
2	34.743	69.487
2	37.891	75.782
4	33.229	66.459
4	53.071	106.142
4	40.407	80.813
4	Not removed from preload	Not removed from preload
5	52.498	104.996
5	58.477	116.953
5	49.756	99.511
5	32.832	65.663
5 (was 4)	45.1238	90.277

An additional set of substrates, labeled 90-day preload, was added to the test matrix (table 5) because of the scatter in the tensile test measurements for the 3-mo control samples. Three sets of three samples each were constructed, and each set of three samples was placed in preload under a separate load. This limited the contact of the sample and load (metal plate and container of lead shot) to three points, ensuring similar loading of each sample, which ensured that minor variations in the height of the assembled samples would not affect the preloading process. This approach eliminated a possible source of variation in the sample processing believed to occur when more than three samples were placed under the same loading (pan/shot/plate) configuration.

Table 5. Bond strength of additional 1-in control samples.

Preload Period (days)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
90 (set 1)	43.595	87.189
90 (set 1)	33.971	67.942
90 (set 1)	25.12	50.24
90 (set 2)	36.019	72.037
90 (set 2)	26.311	52.622
90 (set 2)	33.142	66.284
90 (set 3)	26.196	52.392
90 (set 3)	26.366	52.732
90 (set 3)	36.079	72.158

11. BLOCKING SAMPLES

A set of samples labeled “Blocking Test Samples” was set up to address the scalability of this study’s substrates and load frame measurements obtained from the smaller substrates to an earlier “Blocking Test” conducted by Lockheed-Martin that used 4- by 4-in substrates. The use of primarily 1-in substrates in this study was driven by the need to conserve the supply of MD-944 tape, which is no longer commercially available. The blocking samples were constructed using 1-, 2-, and 4-in-long substrates. These samples, like the control samples, were not exposed to any of the simulated environments. The tensile measurements for the control and the 1-in blocking samples could be combined to increase the statistical accuracy of the study (table 6).

Table 6. Bond strength of blocking samples.

Substrate Length (in)	Preload Period (days)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
1	7	28.079	56.158
1	7	32.665	65.331
1	7	26.623	53.246
1	7	27.286	54.571
1	7	26.937	53.874
1	7	28.929	57.857
1	7	34.19	68.38
1	7	23.052	46.104
2	7	51.861	51.861
2	7	47.116	47.116
2	7	48.158	48.158
4	7	41.258	20.629
4	7	117.819	58.909
4	7	123.093	61.546
4 (set 2)	7	37.347	18.624
4 (set 2)	7	71.754	35.877
4 (set 2)	7	99.295	49.647
4 (set 3)	7	101.276	50.638
4 (set 3)	7	30.958	15.479
4 (set 3)	7	100.465	50.233
4 (set 4)	7	65.813	32.907
4 (set 4)	7	58.887	29.443
4 (set 4)	7	27.702	13.851

Extra sets of 4-in-long substrates were added to the blocking test sample matrix to resolve the variation seen in the tensile strength with the first set. Extra care was taken to minimize bending or twisting that might alter the results.

12. IONIZING RADIATION SAMPLES

The electron radiation energy and fluences for this test were based on radiation environment predictions from the MSFC Environments Branch (EV13) and dose-depth profiling for a 2-mil adhesive layer. Environment predictions were made using the AE-8 electron model and the AP-8 proton model with the design specifications given in SSP 30512 and the as-flown ISS altitude profile, with consideration that the worst-case dose required for this test would be the minimum dose expected, not the maximum dose plus margin. The electrons and protons were produced by Pelletron® accelerators capable of generating 200-keV to 2.5-MeV energy electrons and 30-keV to 700-keV protons.

Samples were exposed to IR at three levels (table 7). The highest level of exposure combined 250-keV electrons (1.15×10^{14} e-/cm² fluence) with 700-keV protons (9.6×10^{10} p+/cm² fluence) while the lower exposure levels (mid IR of 3.9×10^{13} e-/cm², low IR of 1.75×10^{13} e-/cm²) did not include the protons.

Table 7. Bond strength of IR samples.

250-keV Electron Fluence	700-keV Proton Fluence	Preload Duration (days)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
1.15×10^{14}	9.6×10^{10}	7	34.873	69.746
1.15×10^{14}	9.6×10^{10}	7	34.73	69.459
1.15×10^{14}	9.6×10^{10}	7	35.586	71.172
1.75×10^{13}	—	7	24.936	49.871
1.75×10^{13}	—	7	18.366	36.731
1.75×10^{13}	—	7	23.471	46.942
3.9×10^{13}	—	7	22.834	45.668
3.9×10^{13}	—	7	21.331	42.663
3.9×10^{13}	—	7	19.621	39.242

Two additional high-IR sample sets were set up and exposed only to the 250-keV electrons. A set of seven substrates was exposed to the electron fluence, and three samples were constructed using six of those substrates. During the substrate to substrate bonding, one of the substrate pairs was damaged. A second set of eight substrates was set up and exposed to the same electron fluence and then bonded to provide an additional four data points (table 8 and fig. 13).

Table 8. Bond strength of additional high-fluence electron radiation samples.

Electron Fluence	Preload Period (days)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
1.15×10^{14}	7	19.851	39.703
1.15×10^{14}	7	16.527	33.054
1.15×10^{14}	7	19.503	39.006
1.15×10^{14}	7	16.961	33.922
1.15×10^{14}	7	21.824	43.649
1.15×10^{14}	7	20.008	40.017

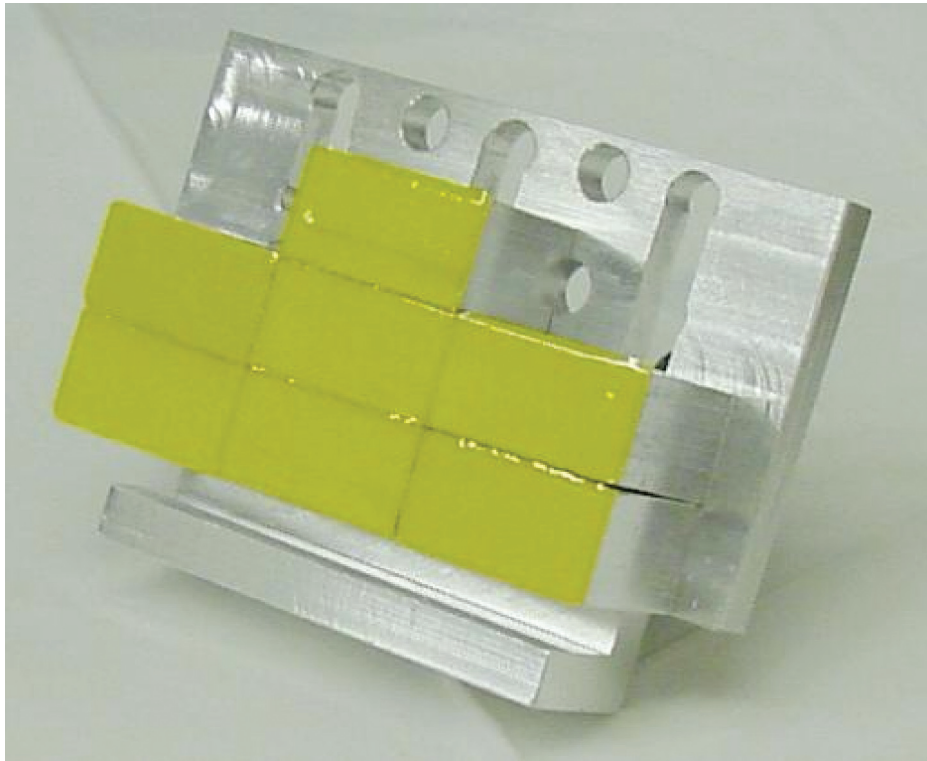


Figure 13. The IR samples loaded in the fixture used during the IR exposures.

13. NEAR ULTRAVIOLET RADIATION SAMPLES

A mercury-xenon lamp was used as the source of the NUV radiation. The samples were mounted in a vacuum chamber and illuminated from outside of the chamber through a UV-transparent port in the side of the chamber. The plate to which the sample holder was mounted was water cooled, and the system operating temperature was 22–30 °C. During the exposure, the chamber was evacuated to a pressure of 10^{-6} torr or less. The lamp was characterized with a radiometer prior to sample exposure, and the output was monitored throughout the test using a photo diode. Figure 14 shows the output from the lamp over the range of 250 to 400 nm.

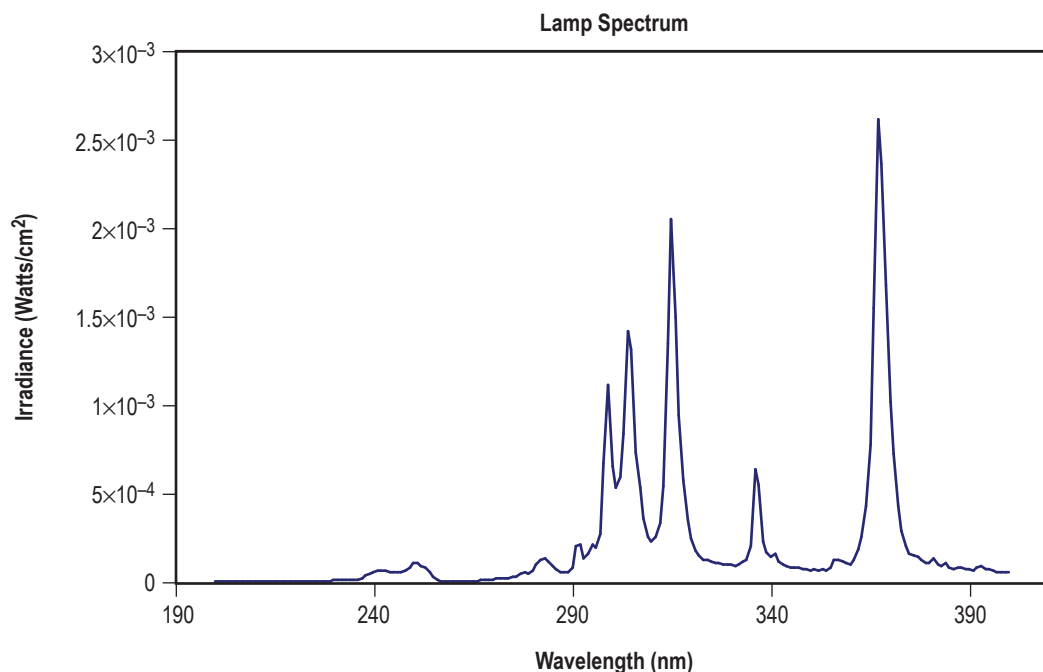


Figure 14. Output of lamp used in NUV radiation exposures.

Three sets of MD-944 tape samples were exposed to NUV for periods of 509, 1,143, and 2,214 equivalent sun hours (ESH) (table 9). The substrates/samples were assembled in the usual manner with the silicone adhesive layer of the MD-944 tape as the top surface. After exposure to NUV, the substrates were assembled into the usual sample pairs and preloaded for 7 days prior to measuring the tensile strength of the silicone-to-silicone bond.

Table 9. Bond strength of NUV-exposed samples.

NUV ESH	Preload Period (days)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
509	7	44.588	89.177
509	7	25.96	51.92
509	7	45.101	90.202
1,143	7	37.796	75.593
1,143	7	23.209	46.417
1,143	7	17.94	35.879
2,214	7	1.385	2.771
2,214*	7	NB**	NB**
2,214	7	26.174	52.347

* Post tensile measurement inspection of bonding surface irregularities in the appearance of the bonding surface. Sample submitted for XPS and FTIR analysis of the bonding surface.

** NB: the samples had no bond formed by the preload.

14. ATOMIC OXYGEN EXPOSURE

Two different facilities were used during the AO exposure of the MD-944 tape samples. The Atomic Oxygen Beam Facility (AOBF) was used to expose samples to 5-eV AO, and the Atomic Oxygen Drift Tube System (AODTS) was used for 0.1-eV AO exposures. The samples exposed in the AOBF facility received a concurrent exposure to vacuum ultraviolet (VUV) radiation, which is a byproduct of AO generation.

Microcracking was apparent on samples exposed to AO (fig. 15) and also on samples exposed to a combination of AO, NUV, and IR. This is consistent with silicate formation on the surface. Samples exposed to less AO had similar but fewer cracks. Samples exposed to NUV or IR with no AO did not exhibit microcracking.

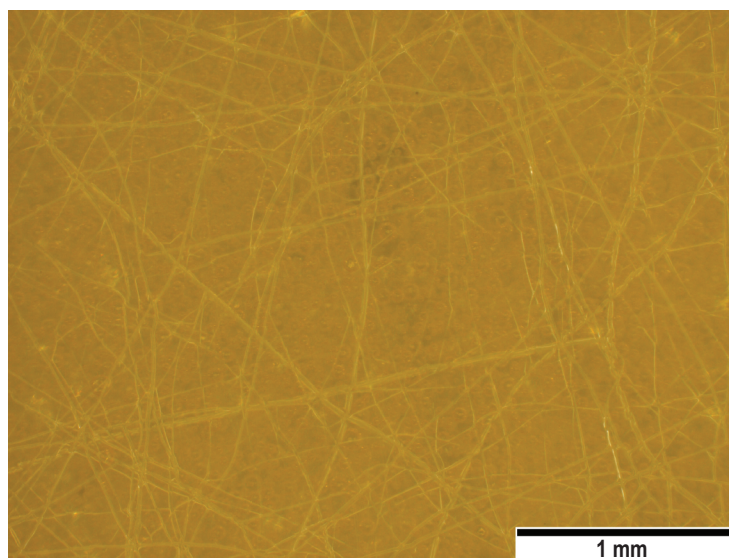


Figure 15. Photomicrograph of sample exposed to 5-eV AO fluence of 1.40×10^{21} atoms/cm².

15. ATOMIC OXYGEN BEAM FACILITY SAMPLES

The tensile testing data for the AOBF samples (fig. 16) and the exposure parameters are listed in table 10.

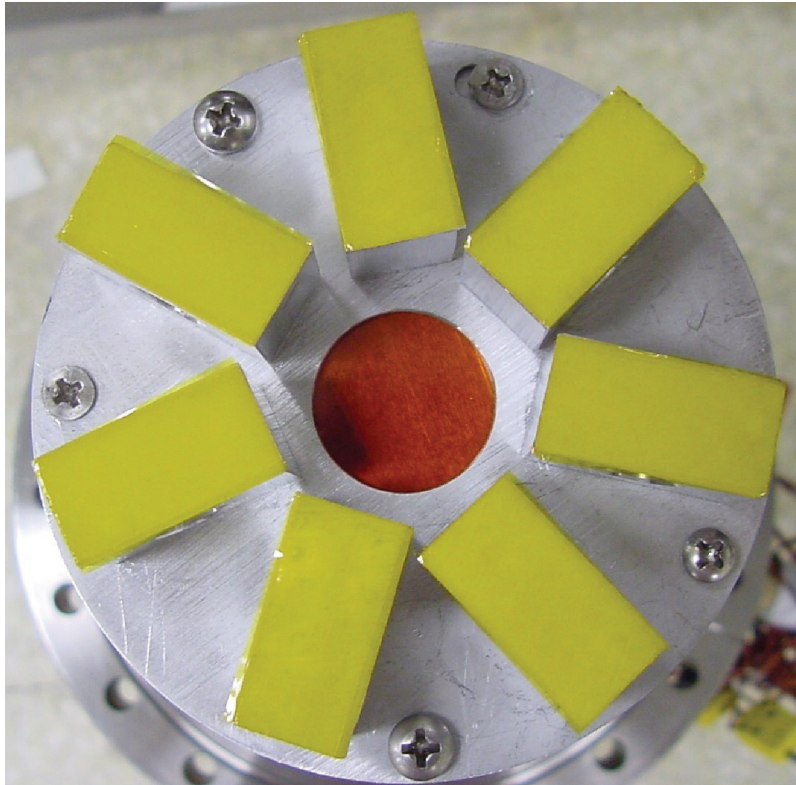


Figure 16. Seven substrates mounted on the AOBF sample holder. The orange-brown disk at the center of the picture is a Kapton film sample used to monitor the fluence of AO to the sample.

Table 10. Bond strength of samples exposed to 5-eV AO.

Preload Period (days)	AO Fluence	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
7	1.3×10^{18}	25.644	51.287
7	1.3×10^{18}	33.842	67.638
7	1.3×10^{18}	25.681	51.363
7	3.0×10^{19}	8.941	17.882
7	3.0×10^{19}	5.836	11.672
7	3.0×10^{19}	9.682	19.365
7	9.7×10^{19}	0.882	1.763
7	9.7×10^{19}	0.117	0.234
7	9.7×10^{19}	NB*	NB*
7	1.4×10^{21}	NB*	NB*
7	1.4×10^{21}	NB*	NB*
7	1.4×10^{21}	NB*	NB*

* NB: the samples had no bond formed by the preload.

Some samples were sequentially exposed to 5-eV AO, NUV radiation and ionizing radiation. The set labeled “Reverse” had IR first, then NUV, then 5-eV AO. Table 11 contains the tensile measurements for those samples.

Table 11. Bond strength of samples exposed to multiple simulated environments.

Preload Period (mo)	AO Fluence	NUV Fluence (ESH)	IR Fluence (e-/cm ²)	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
3	1.18×10^{21}	2,687.1	1.75×10^{13}	NB*	NB*
3	1.18×10^{21}	2,687.1	1.75×10^{13}	NB*	NB*
3	1.18×10^{21}	2,687.1	1.75×10^{13}	0.309	0.619
9.58	1.42×10^{21}	2,613.9	1.75×10^{13}	<<1**	<<1**
9.58	1.42×10^{21}	2,613.9	1.75×10^{13}	<<1**	<<1**
9.58	1.42×10^{21}	2,613.9	1.75×10^{13}	<<1**	<<1**
17	1.48×10^{21}	2,214	N/A	<<1**	<<1**
17	1.48×10^{21}	2,214	N/A	<<1**	<<1**
17	1.48×10^{21}	2,214	N/A	<<1**	<<1**
3—Reverse	4.27×10^{20}	2,180	1.73×10^{13}	NB*	NB*
3—Reverse	4.27×10^{20}	2,180	1.73×10^{13}	NB*	NB*
3—Reverse	4.27×10^{20}	2,180	1.73×10^{13}	NB*	NB*

* NB: The samples had no bond formed by the preload.

** <<1: The samples did form a bond during preload, but the bond failed during sample handling or under the weight of the sample pair.

16. ATOMIC OXYGEN DRIFT TUBE SYSTEM SAMPLES

The tensile test measurements for the samples exposed to thermal energy (generally <0.1 eV) AO in the AODTS are presented in table 12. Unlike the AOBF system, no exposure of the samples to VUV radiation occurs in this system.

Table 12. Bond strength of samples exposed to thermal energy AO.

Preload Period (days)	AO Fluence	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
7	1.53×10^{22}	6.191	12.382
7	1.53×10^{22}	0.581	1.163
7	1.53×10^{22}	0.386	0.771
7	2.24×10^{22}	NB	NB
7	2.24×10^{22}	NB	NB
7	2.24×10^{22}	<<1	<<1
7	2.3×10^{22}	0.14	0.28
7	2.3×10^{22}	0.312	0.625
7	2.3×10^{22}	0.084	0.169
7	2.404×10^{22}	NB	NB
7	2.404×10^{22}	0.084	0.168
7	2.404×10^{22}	0.272	0.543
30*	3.6×10^{22}	<<1	<<1
30*	3.6×10^{22}	<<1	<<1
30*	3.6×10^{22}	<<1	<<1

* The bond between the substrates failed while the sample was being loaded into the test fixture.

17. X-RAY PHOTOELECTRON SPECTROSCOPY ANALYSIS

Several of the samples were analyzed by x-ray photoelectron spectroscopy (XPS) with the objective of detecting a transition between the chemistry of the surface of the adhesive and the underlying layers from exposure to AO, IR, VUV, and NUV radiation. The outer surface of the adhesive, a polydimethyl polysiloxane, should be transformed into glass-like silicate by exposure to AO and the depth or thickness of the silicate may increase with increasing AO fluence.

XPS analysis of the surface of the adhesive did detect a change in the bonding energy of the silicone adhesive from a nominal 101 eV for the silicone to a nominal 103 eV for the silicate. This change in the adhesive was not detected in the adhesive layer of control samples but was detected in the surface of adhesive exposed to AO and NUV exposed samples. Appendix B, Report on the ESCA Evaluation of MD-944 Diode Tape, contains a detailed description of the XPS analysis. The terms XPS and electron spectroscopy for chemical analysis (ESCA) are different names for the same analytical technique and are applied interchangeably in this TM. It should be noted that the presence of tantalum in the XPS analysis is due to the use of a tantalum neutralizer plate in creating the 5-eV AO beam.

18. FOURIER TRANSFORM INFRARED SPECTROSCOPY ANALYSIS

The absorbance peak at $1,250\text{ cm}^{-1}$ in figure 17 indicates the presence of the silicone adhesive. This peak is significantly reduced in figure 18, indicating the silicone adhesive has been altered by the AO and NUV exposure. None of the FTIR analyses, appendix C, detected a significant absorbance band at $1,400\text{ cm}^{-1}$ (Si-CH_3) and the broad absorbance band at $1,110\text{--}1,100\text{ cm}^{-1}$ did not provide a clear indication of the conversion of the silicone adhesive to a silicate (SiO_x). Even the FTIR scans of the AO exposed sample do not show any clear evidence of a silicate absorbance peak.

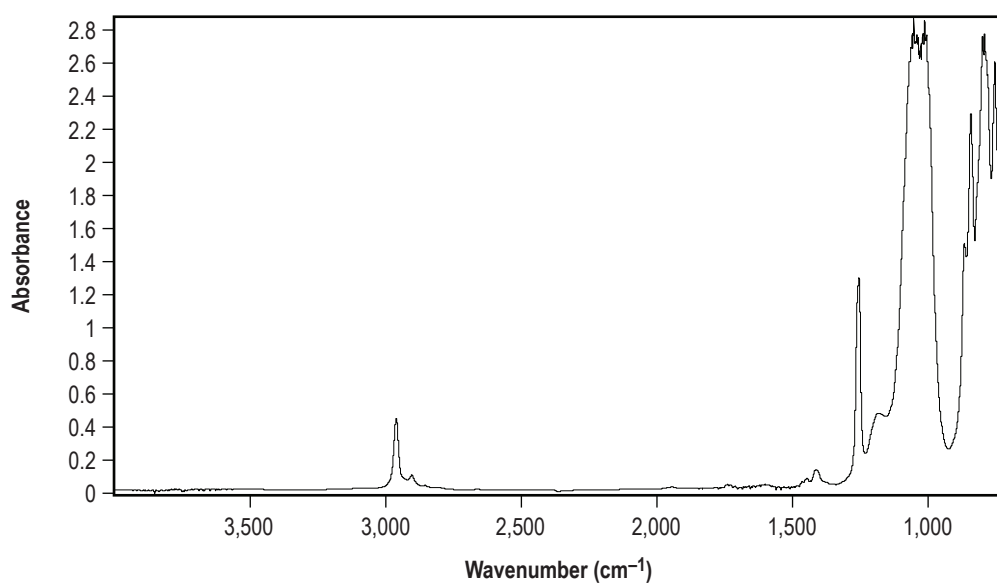


Figure 17. MD-944 tape control sample No. 133 (C1C).

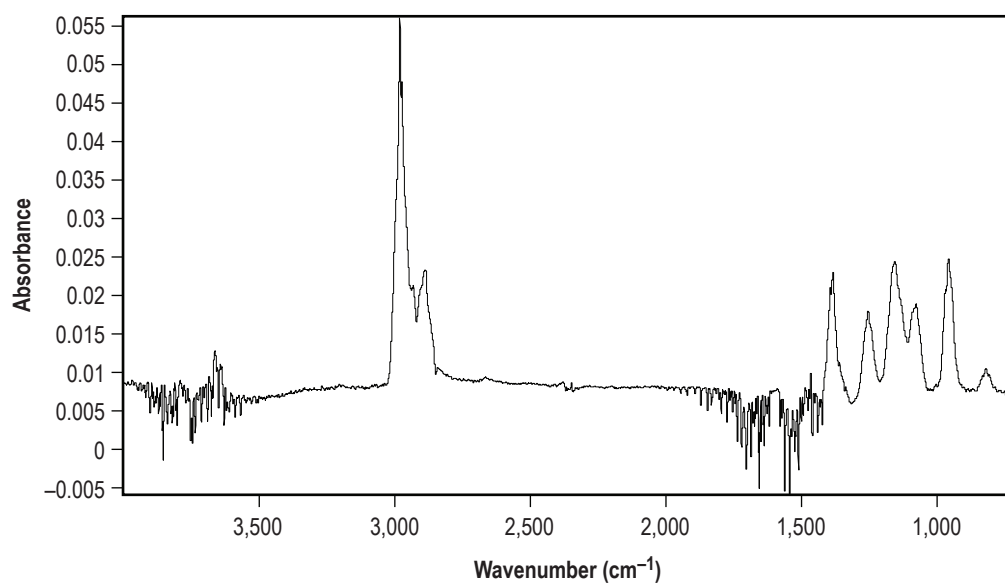


Figure 18. AOBf-BF1 sample No. 163 (1.48×10^{21} atoms/cm²) with 2,200-ESH.

19. SUMMARY OF RESULTS

The environmental factors found in low Earth orbit will reduce the bond strength of the MD-944 silicone adhesive below the 2.5-psi mechanical force provided by the ISS solar array deployment mechanism. NUV and AO were effective in reducing the bond strength of the MD-944 diode tape adhesive through a chemical change or other degradation mechanism. The role of VUV in the degradation of the adhesive, during the 5-eV AO exposures of the adhesive in the AOBF, was not clearly demonstrated. Exposure of the adhesive to thermal energy AO did result in a clear reduction in the performance of the adhesive and does not include the concurrent VUV exposure of the 5-eV process. Exposure of the adhesive layer to NUV did result in a clear degradation of the performance of the adhesive bond strength but to a lesser degree than the exposure to AO. Exposure of the adhesive to 250-keV electrons produced very little degradation in the performance of the silicone adhesive. Adhesive sequentially exposed to AO, NUV and IR also resulted in a significant degradation in the strength of the adhesive bond. Lowering of the adhesive bond strength is due in part to the chemical conversion of the silicone adhesive to a silicate or glass-like material. However, other degradation processes are not ruled out, and the chemistry of the adhesive degradation is likely a complex process.

Exposure of the adhesive to 9.0×10^{19} atoms/cm² AO (5 eV), 1.5×10^{22} atoms/cm² thermal energy AO (<0.1 eV), or more than 2,200 ESH NUV was demonstrated to lower the bond strength of the adhesive to below 2.5 psi. Since the P6 solar array has been deployed (November 2000), it has received in excess of 1×10^{22} atoms/cm² of orbital AO and 10,000 ESH of solar UV. Any exposed silicone adhesive should be fully converted to silicate, with minimal stiction.

APPENDIX A—KAPTON-UP ANALYSIS

Two sample sets were constructed with the Kapton layer of the MD-944 tape in a typical as-applied configuration where the silicone adhesive bonds to the substrate and the Kapton layer is exposed to the environment. These samples were exposed to 5-eV AO in the AOBF to demonstrate the degradation of the silicone adhesive layer after erosion of the Kapton layer by AO. The first sample sets (table 13) were constructed by applying the silicone adhesive layer directly to the PSA layer. The second set of samples (table 14) was constructed without the PSA layer, and the silicone adhesive of the MD-944 tape was bonded directly to the aluminum substrate.

Table 13. Bond strength of Kapton-up samples with PSA layer exposed to 5-eV AO.

Preload Period (days)	AO Fluence	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
7	4.2×10^{20}	0.094	0.189
7	4.2×10^{20}	NB	NB
7	4.2×10^{20}	NB	NB
7	4.5×10^{20}	NB	NB
7	4.5×10^{20}	NB	NB
7	4.5×10^{20}	<<1*	<<1*
7	4.9×10^{20}	NB	NB
7	4.9×10^{20}	<<1*	<<1*
7	4.9×10^{20}	<<1*	<<1*
7	1.18×10^{21}	NB	NB
7	1.18×10^{21}	NB	NB
7	1.18×10^{21}	NB	NB

* The bond between the substrates separated before the sample could be loaded in the load frame fixture.

Table 14. Bond strength of Kapton-up samples without additional PSA layer exposed to 5-eV AO.

Preload Period (days)	AO Fluence	Load at Maximum Load (lbf)	Stress at Maximum Load (psi)
7	4.27×10^{20}	0.862	1.724
7	4.27×10^{20}	0.477	0.953
7	4.27×10^{20}	0.242	0.484
7	2.1×10^{21}	NB	NB
7	2.1×10^{21}	NB	NB
7	2.1×10^{21}	NB	NB
7	4.27×10^{20}	NB	NB
7	4.27×10^{20}	1>>	1>>
7	4.27×10^{20}	1>>	1>>

A.1 Atomic Oxygen Erosion of Kapton Bonded Directly to the Aluminum Substrate

Halfway through the AO exposure of sample set KU5, the samples were removed from the AOBF and visually examined. At that time, the Kapton layer was fully eroded away and the silicone adhesive could be observed as a layer on top of the aluminum substrate (fig. 19). The samples were then placed back in the AOBF to complete the AO exposure shown in table 14.

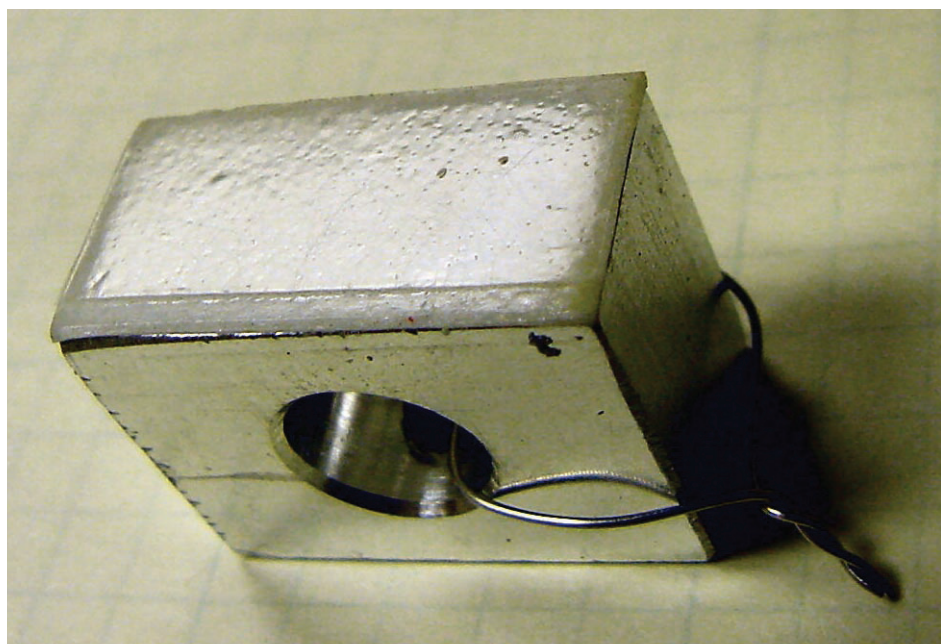


Figure 19. Kapton layer shown eroded away and the silicone adhesive observed as a layer on top of the aluminum substrate.

APPENDIX B—REPORT ON THE ESCA EVALUATION OF MD-944 DIODE TAPE

Analysis by Dr. Binayak Panda, Marshall Space Flight Center (EM30)

The MD-944 Diode Tape contained a silicone adhesive with a chemical formula poly- (dimethyl siloxane) or PDMS. The effectiveness of the silicone adhesive could be impaired due to its exposure to atomic oxygen, photons such as x-rays and ultraviolet light as encountered in space. For several samples exposed in such a manner, electron spectroscopy (ESCA) was used to evaluate such degradation of the adhesive. It is worth mentioning that exposure to x-rays during evaluation in ESCA could also degrade the adhesive. Therefore, to minimize such degradation shorter exposure was planned during the initial analysis of the tape. Following this initial analysis, for some samples, the analyzed region was exposed to x-rays for several hours inside ESCA and then analyzed to see if there was a significant degradation during the initial analysis. In this report, only the initial results have been reported since the effects of x-ray degradation were minimal.

Besides H, PDMS has three ingredients: Carbon (C), silicon (Si) and oxygen (O). It is known, for this material that the C/Si ratio decreases with increase in time of exposure to radiation. It is possible that radiation would transform silicone to silicate. Such a transformation would indicate a higher Binding Energy (BE) for Si 2p line. If this happens, there would be two Si 2p peaks of silicon — one for silicone (BE around 101 eV) and the other for silicate (BE around 103 eV). It was, therefore, planned that samples be evaluated with reference to an unexposed (to radiation) sample. The ESCA evaluation results were geared toward the following.

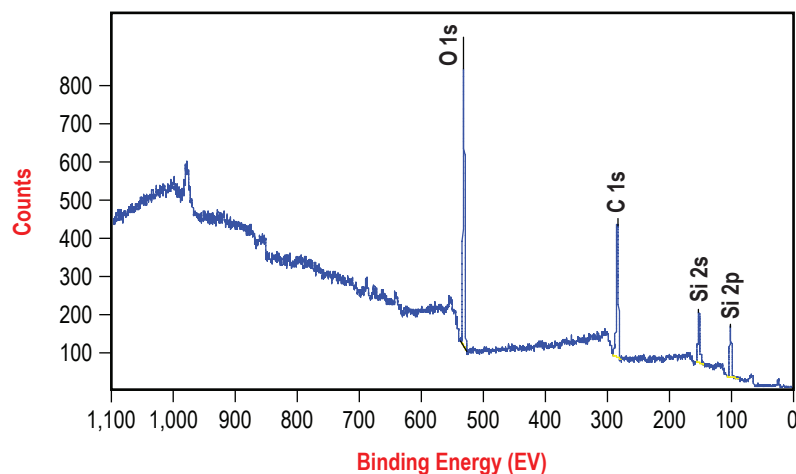
- (1) An overall scan to identify all elements or any contamination present on the surface.
- (2) Regional scans to show the O, C, and Si peaks to measure their intensity. Any peak shift or change area under a peak would indicate degradation and/or formation of another compound.
- (3) Ratio of C/Si intensities of the peaks to assess degradation due to radiation exposure.

The samples were mounted on a special holder and a fine nickel screen with 90 percent transmission was placed on top of the sample to dissipate electrons generated from charging effects. A flood gun was also used simultaneously for the same purpose for better effectiveness. Despite these efforts, peaks were broad and were skewed somewhat. For any peak position measurement or shifting due to charging, O 1s peak was taken as reference at 532.0 eV for all samples analyzed.

Sample No. 188 represented the original PDMS material with no exposure to any radiation. Figure 20 shows C, O, and Si in its spectrum. To illustrate the shapes of the peaks, figures 21, 22, and 23 are presented from a set of regional scans for the sample. It is clear from these figures that the peaks are somewhat wider and a bulge on each peak exists to the right of each peak. While this can be attributed to the uneven charging (differential charging) within the x-ray spot, the total area of the peak and the position of the main peak are utilized in the analysis for the peak intensity and the line positioning, respectively. It is important to point out that instead of peak fitting if the whole peak is assumed as one,

the estimated Si 2p position would be different than what would be obtained from the peak fitting exercise. For the silicone to convert to silicate the line shift could be noted and often times the Si peak could be resolved into two peaks (see fig. 24). The intensity ratio of these two peaks is reported as % Silicate Conversion in table 15.

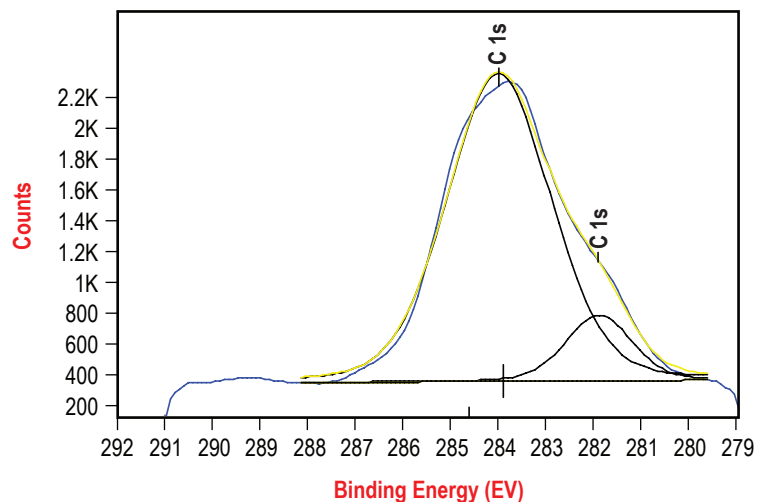
Project: 2003-829		Acquired: 12:00:00 AM by Administrator	
Experiment: 188-02		Description: Overall scan, CN at Minimum	
Region: Regn0	Res: 3	Etch Time:	
Scns/Time: 2	Spot: 3	Cycle No.:	



XPS Line	Adj'd Be	CrossSec	Norm Area	Atom %
O 1s	531.733	2.93	212.006	16.455
C 1s	283.95	1	557.386	43.262
Si 2p	100.976	0.817	518.994	40.282

Figure 20. An overall spectrum of undamaged PDMS showing O, C, and Si peaks as the main constituents. Sample No. 188.

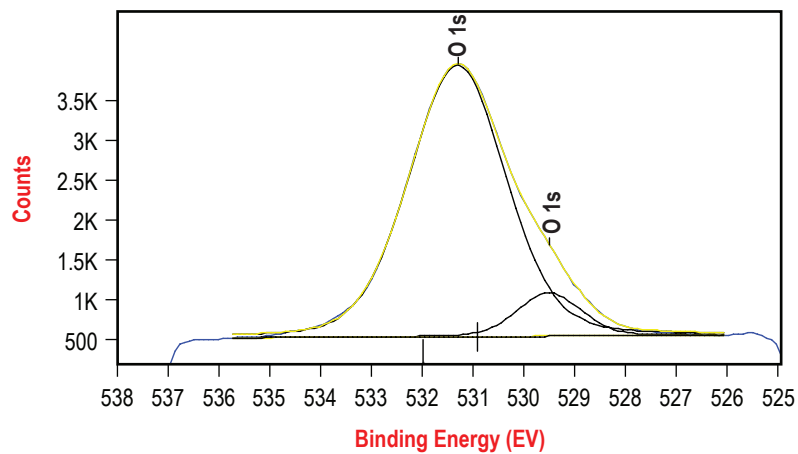
Project: 2003-829 Experiment: 73-07		Acquired: 12:00:00 AM by Administrator Description: Experiment Comment	
Region: Regn0 Scns/Time: 119.6 s	Res: 3 Spot: 3	Etch Time: Cycle No.:	



XPS Line	Adj'd Be	CrossSec	Norm Area	Atom %
C 1s	283.968	1	496.263	87.759
C 1s	281.876	1	69.22	12.241

Figure 21. Shows the C 1s peak which can be split into 2 peaks. Position and intensities of each is shown. The smaller peak to the right in this and other figures could be due to differential charging. Sample No. 73.

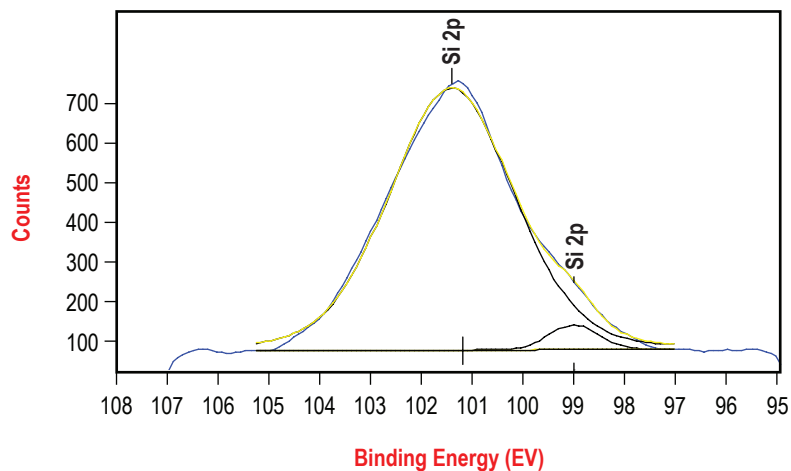
Project: 2003-829 Experiment: 73-07		Acquired: 12:00:00 AM by Administrator Description: Experiment Comment
Region: Regn0 Scns/Time: 119.7 s	Res: 3 Spot: 3	Etch Time: Cycle No.:



XPS Line	Adj'd Be	CrossSec	Norm Area	Atom %
O 1s	531.301	2.93	163.935	90.437
O 1s	529.498	2.93	17.335	9.563

Figure 22. Oxygen peaks in PDMS. The peak on the right could be due to differential charging. Sample No. 73.

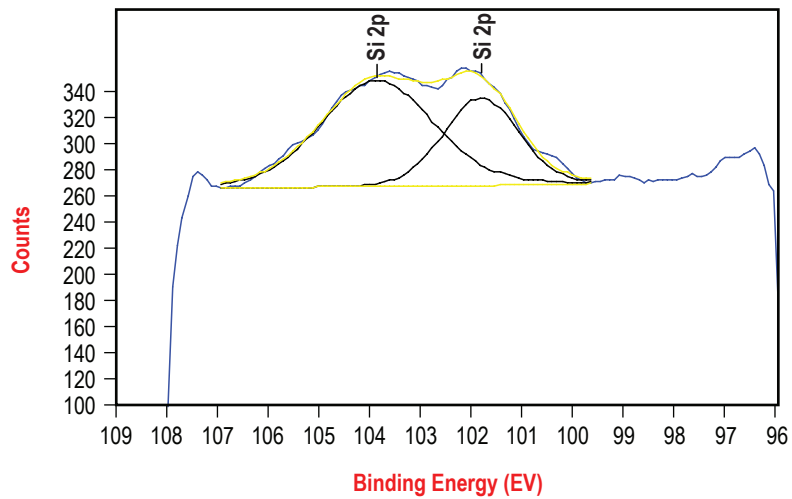
Project: 2003-829 Experiment: 73-07		Acquired: 12:00:00 AM by Administrator Description: Experiment Comment	
Region: Regn0 Scns/Time: 119.5 s	Res: 3 Spot: 3	Etch Time: Cycle No.:	



XPS Line	Adj'd Be	CrossSec	Norm Area	Atom %
Si 2p	101.404	0.817	465.833	96.167
Si 2p	99.000	0.817	18.566	3.833

Figure 23. Si 2p Peaks in Si. The small peak to the right is due to charging effects. As the silicate forms, a second peak may appear to the left of the larger peak. Sample No. 73.

Project: 2003-829		Acquired: 12:00:00 AM by Administrator	
Experiment: 73-07		Description: Regional for Degrd. Single Scan, CN at Minimum	
Region: Regn0	Res: 3	Etch Time:	
Scns/Time: 59.6 s	Spot: 3	Cycle No.:	



XPS Line	Adj'd Be	CrossSec	Norm Area	Atom %
Si 2p	103.852	0.817	102.622	63.865
Si 2p	101.799	0.817	58.063	36.135

Figure 24. In sample No. 78 silicate peak appears on the left at a higher BE region.

Table 15. Summary of peak intensity and line position results.

Sample No.	Peak	Peak Position	Corrected Peak Position	% Silicate Conversion	Peak Intensity
188	C 1s	284	284.5	–	516.5
–	O 1s	531.5	532	–	339.7
–	Si 2p	101.3	101.8	0	213.2
73	C 1s	284.8	285.4	–	335.3
Ta, F present	O 1s	531.4	532	–	601.2
–	Si 2p *	103.8	104.4	64	98.5
197	C 1s	284.9	284	–	518.5
–	O 1s	532.9	532	–	356.3
–	Si 2p *	103	102.1	18	119.1
146	C 1s	284.8	283.8	–	388 **
F present	O 1s	533	532	–	744.3 **
–	Si 2p *	103.3	102.3	71	324.8 **
171	C 1s	284.5	283.8	–	347.1
F present	O 1s	532.7	532	–	540.2
–	Si 2p *	101.8	101.1	38	256.9
–	C 1s	284.8	285.5	–	435.9
123	O 1s	531.3	532	–	344.3
Ta and some F	Si 2p	102.7	103.4	79	85.5
95	C 1s *	286.6	284.1	–	239.8
F and some Ta	O 1s *	534.5	532	–	509.1
–	Si 2p	105.1	102.6	83	141.9
202	C 1s	284.4	283.9	–	268.8
F present	O 1s	532.5	532	–	579
–	Si 2p *	101.4	100.9	45	225.9
163	C 1s	284.7	284.9	–	439.5
Ta and some F	O 1s	531.8	532	–	536.7
–	Si 2p *	102.8	103	63	141.2
166	C 1s	284.8	285.2	–	376.9
Some F present	O 1s *	531.6	532	–	629.8
–	Si 2p *	101.9	102.3	44	283.6

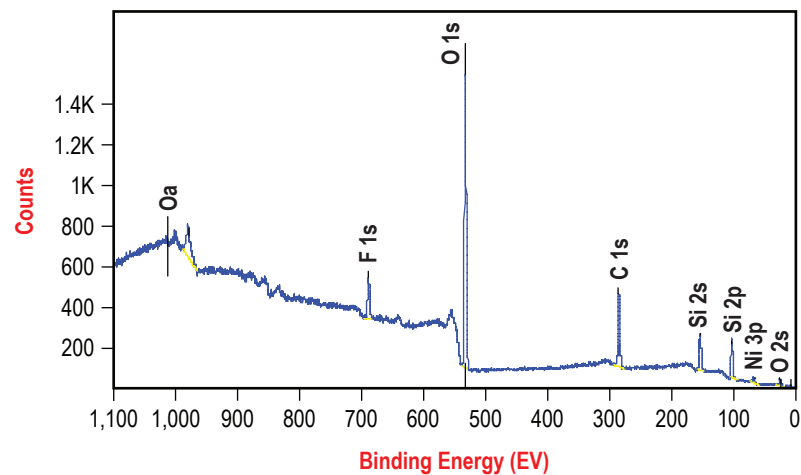
* Indicates that there are two peaks of significant intensity.

** 'Scanned' spectra was used in place of 'unscanned' data to evaluate; absolute intensity is different from the rest.

Table 15 lists the sample numbers analyzed, and the peak positions and intensities for the C, O, and Si peaks. The Peak Position in table 15 corresponds to the main peak positions evaluated on the spectrum. Under one peak, say C 1s, there may be several and they can be separated through curve fitting, and 'Peak Position' indicates the position of the most prominent of them all. The Corrected Peak Position refers to the position when the main peak is shifted to a new position as the O 1s peak is set at 532.0 eV. The intensity of the peak is simply the total area under the curve for a peak.

For each sample, C and Si intensity ratios could be calculated easily from the Peak Intensity column of table 15. But, the results are not reported as no correlation could be found between this degradation and photonic or ionic exposure. Contaminations such as flourine (F) and tantalum (Ta) were found on the sample surfaces are reported in the sample column in table 15. Figures 25–27 show the presence and intensity of some of the samples.

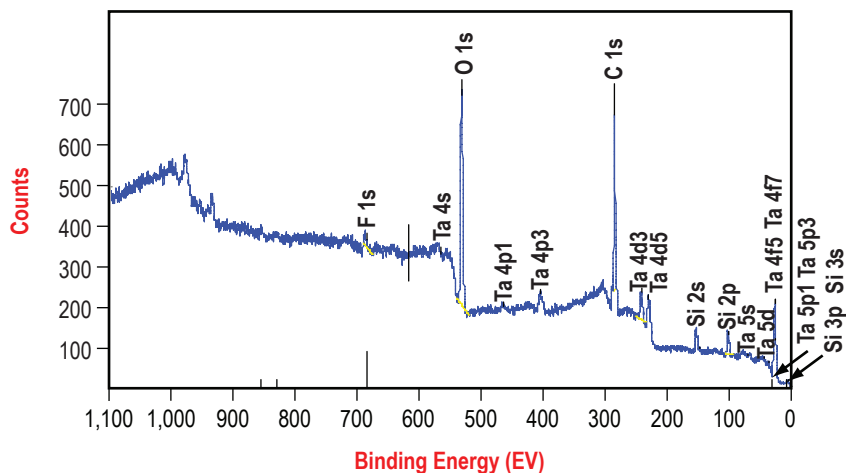
Project: 2003-829		Acquired: 12:00:00 AM by Administrator	
Experiment: 146-02		Description: Overall-CN at Minimum	
Region: Regn0	Res: 3	Etch Time:	
Scns/Time: 2	Spot: 3	Cycle No.:	



XPS Line	Adj'ed Be	CrossSec	Norm Area	Atom %
O 1s	532.942	2.93	422.977	28.836
F 1s	688.660	4.43	38.577	2.630
C 1s	285.077	1	443.794	30.255
Si 2s	153.800	0.955	507.730	34.613
Ni 3p	67.554	2.217	53.782	3.666

Figure 25. F found on the surface of sample No. 146.

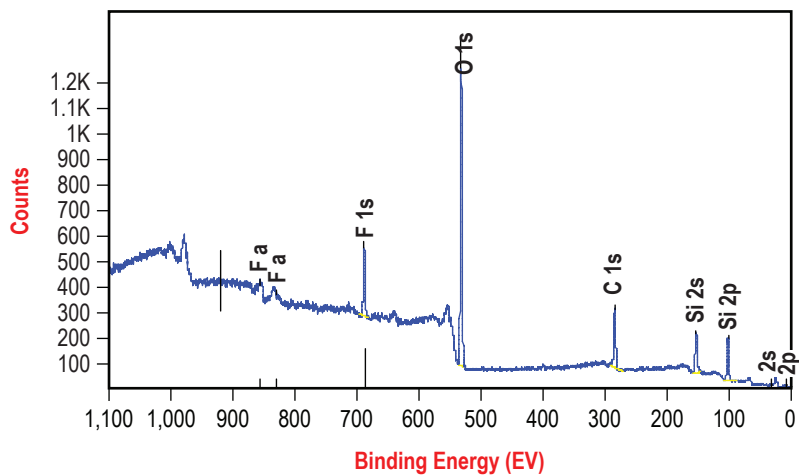
Project: 2003-829		Acquired: 12:00:00 AM by Administrator
Experiment: 123-01		Description: NS, All-in-One, CN at Minimum
Region: Regn0	Res: 3	Etch Time:
Scns/Time: 2	Spot: 3	Cycle No.:



XPS Line	Adj'd Be	CrossSec	Norm Area	Atom %
C 1s	284.979	1	442.667	51.066
O 1s	531.710	2.93	205.343	23.688
Ta 4d3	242.519	6.4	19.131	2.207
Si 2p	101.758	0.817	191.776	22.123
F 1s	685.422	4.43	7.934	.915

Figure 26. F and Ta found on surface of sample No. 123. Si peaks are much smaller compared to C and O.

Project: 2003-829		Acquired: 12:00:00 AM by Administrator	
Experiment: 202-01		Description: NS, All-in-One, CN at Minimum	
Region: Regn0	Res: 3	Etch Time:	
Scns/Time: 2	Spot: 3	Cycle No.:	



XPS Line	Adj'd Be	CrossSec	Norm Area	Atom %
O 1s	531.917	2.93	344.546	26.685
F 1s	688.002	4.43	41.650	3.226
C 1s	284.441	1	336.549	26.066
Si 2p	101.993	0.817	568.404	44.023

Figure 27. F on the surface of sample No. 202.

APPENDIX C—ADDITIONAL FOURIER TRANSFORM INFRARED SPECTRA

FTIR spectroscopy was used to look at several samples (figs. 28–38) in addition to those reported in the FTIR analysis section of this TM. As reported in that section, the absorbance peak at $1,250\text{ cm}^{-1}$ in the spectra indicates the presence of the silicone adhesive. This peak is significantly reduced in some of the figures, indicating that the silicone adhesive has been altered by the AO and NUV exposure. None of the FTIR analyses detected a significant absorbance band at $1,400\text{ cm}^{-1}$ (Si-CH_3), and the broad absorbance band at $1,110\text{--}1,100\text{ cm}^{-1}$ did not provide a clear indication of the conversion of the silicone adhesive to a silicate (SiO_x). Even the FTIR scans of the AO exposed sample do not show any clear evidence of a silicate absorbance peak.

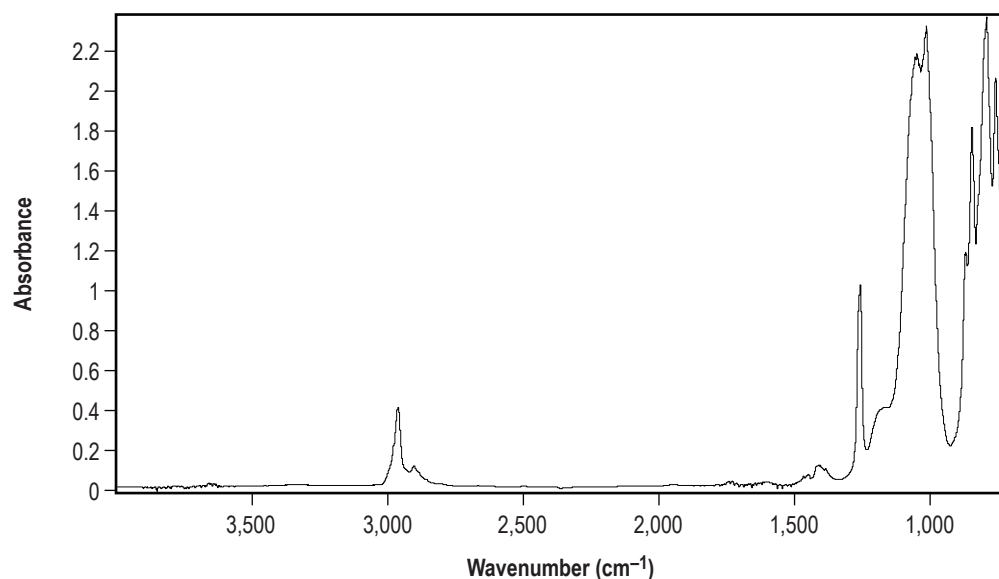


Figure 28. IR sample No. 81 high electron dose (250 keV , 1.15×10^{14} fluence) without protons.

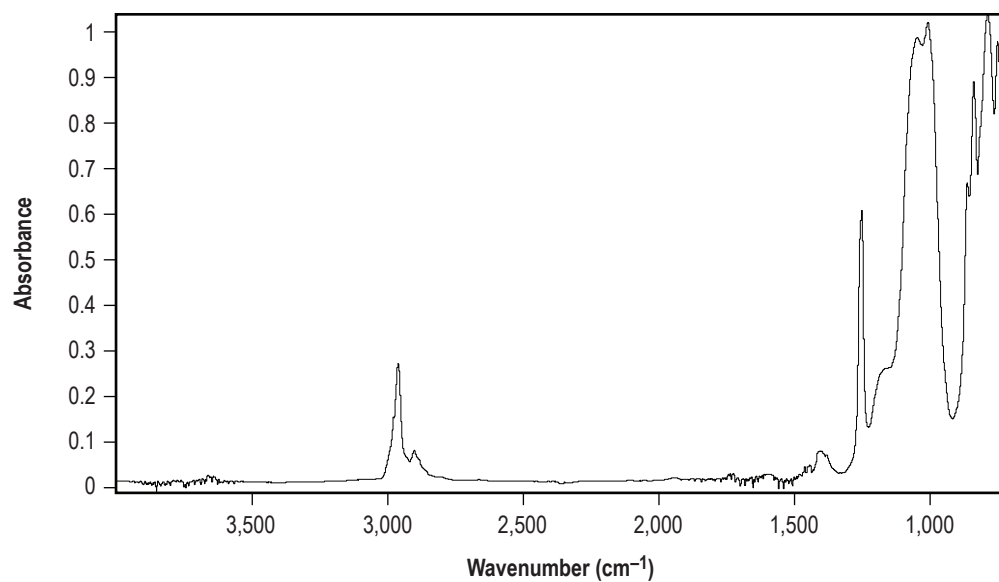


Figure 29. IR sample No. 162 mid dose (250 keV, 3.9×10^{13} fluence) electrons.

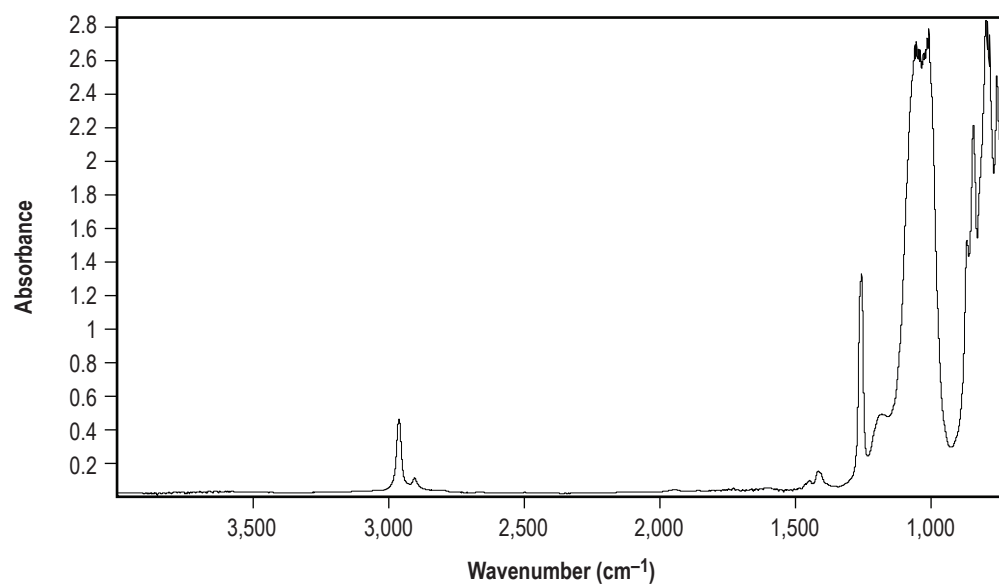


Figure 30. IR sample No. 125 low dose (250 keV, 1.75×10^{13} fluence) electrons.

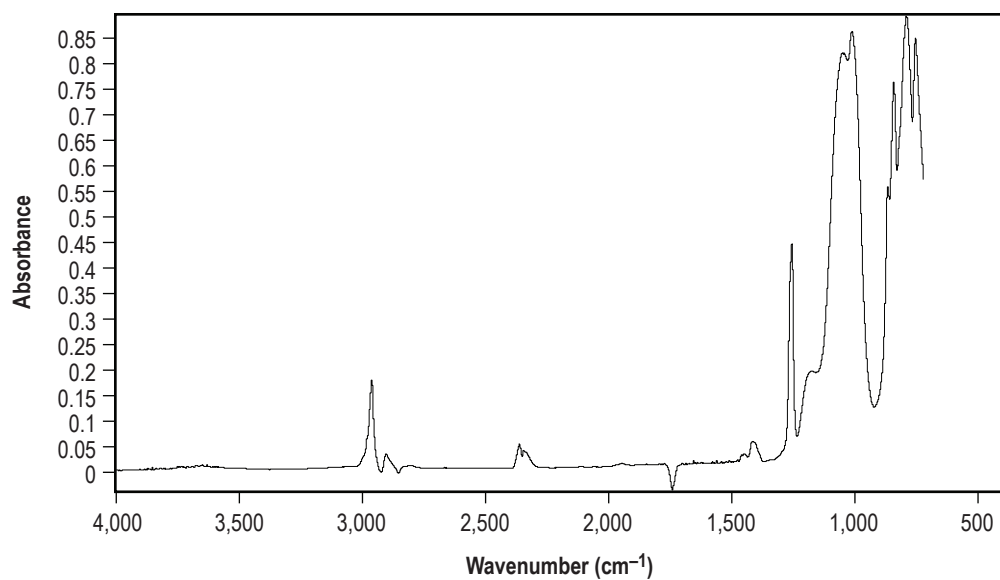


Figure 31. IR sample No. 25, (250 keV, 1.15×10^{14} fluence) electrons with (700 keV, 9.6×10^{10} fluence) protons.

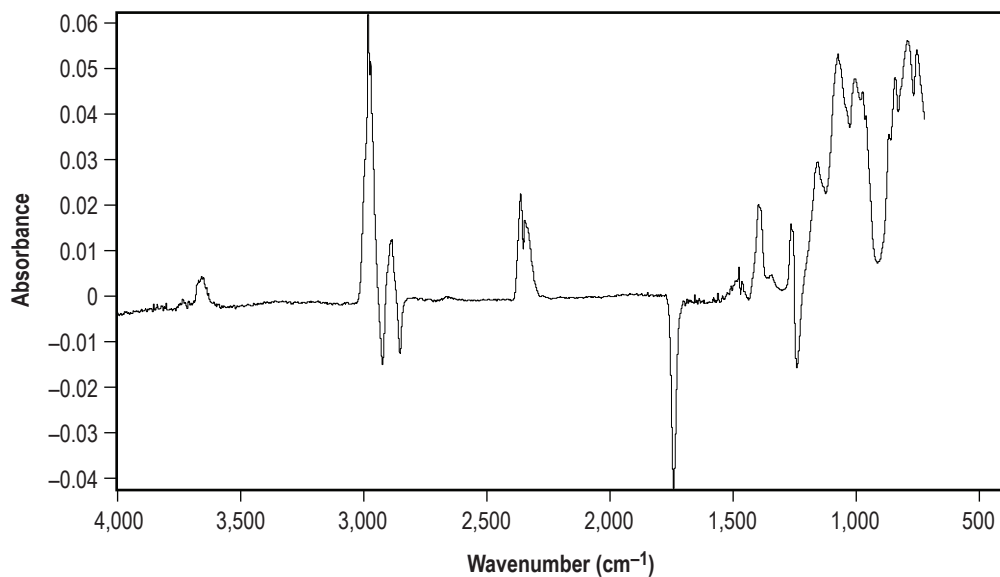


Figure 32. IR sample No. 25 with protons, scan of a different area of the tape.

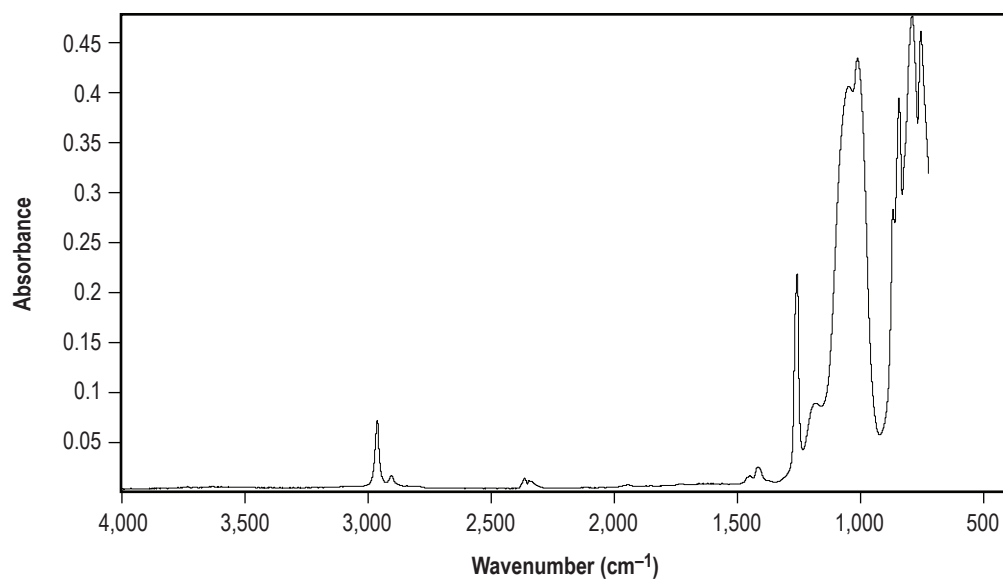


Figure 33. NUV sample No. 174, 500-ESH.

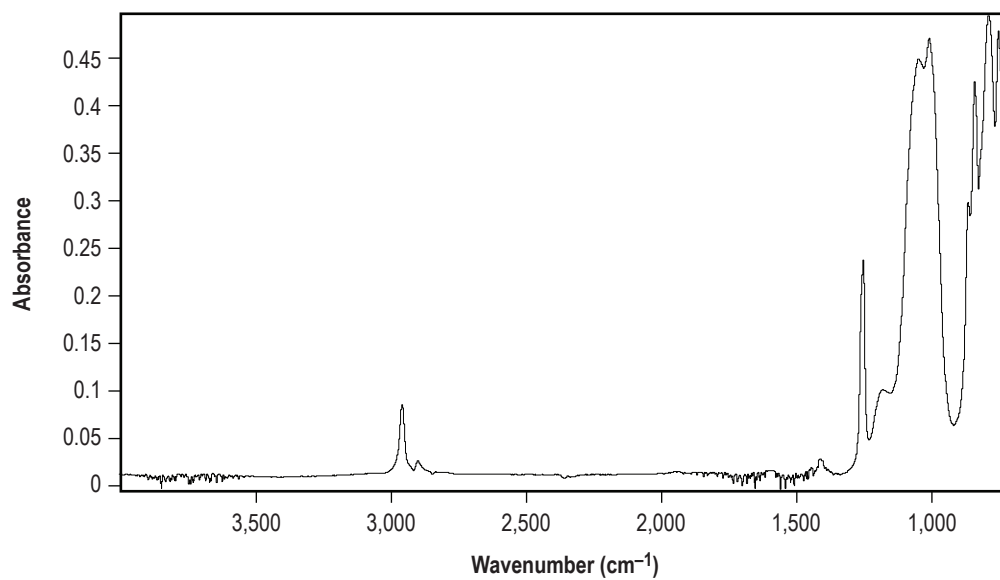


Figure 34. NUV sample No. 120, 1,100-ESH.

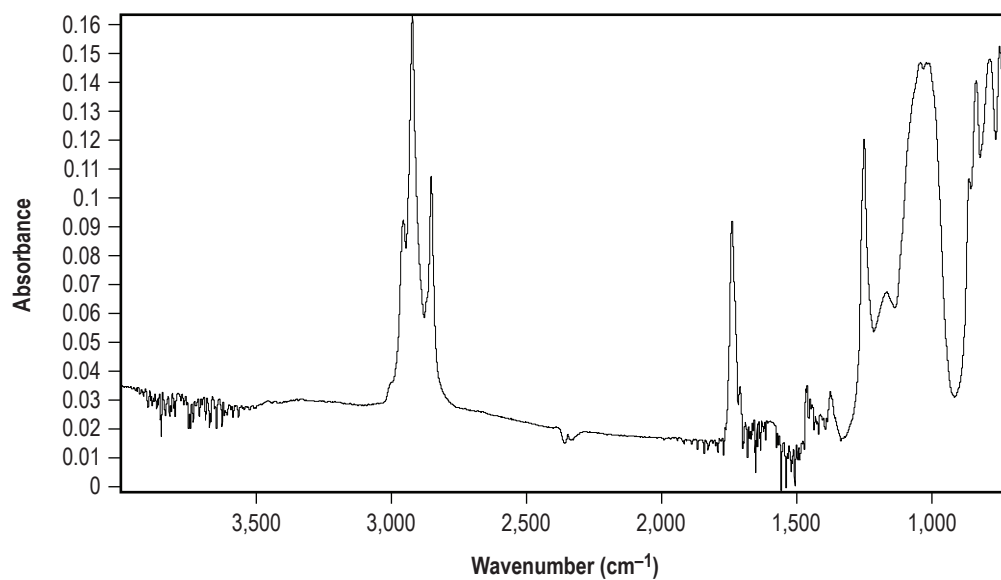


Figure 35. NUV sample No. 124, 2,200-ESH.

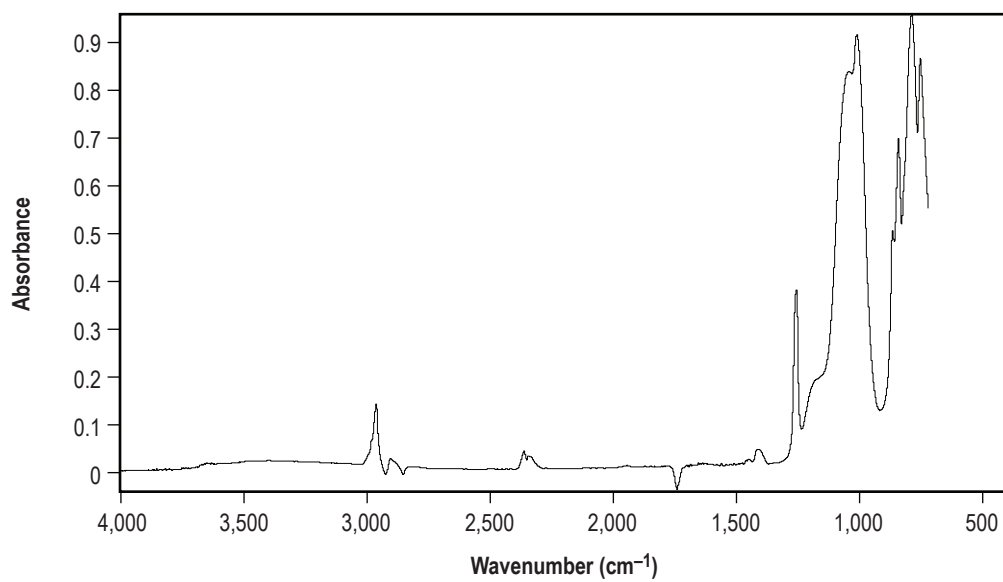


Figure 36. AOBFBF₄ sample No. 95, (9.7×10^{19} atoms/cm²).

C.1 Supplementary Spectra

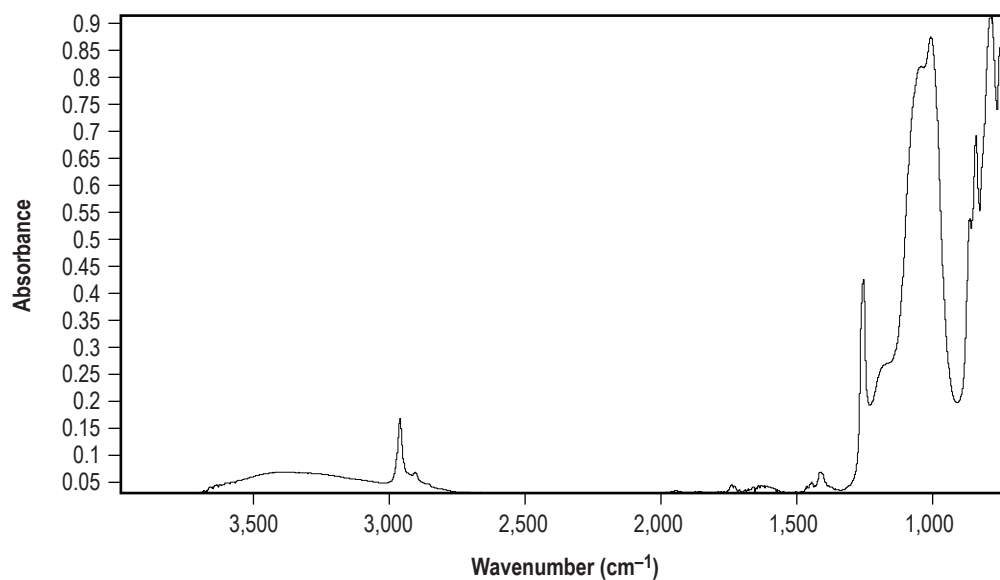


Figure 37. AOBf-BF₄ sample No. 171 (9.7×10^{19} atoms/cm²), after tensile test.

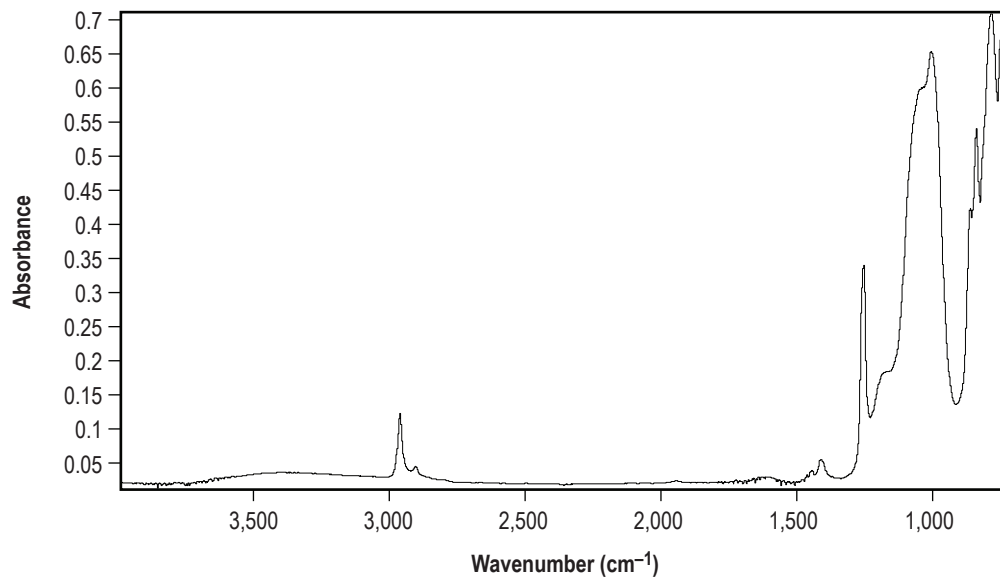


Figure 38. AOBf-BF₄ sample No. 128 (5 eV, 9.7×10^{19} atoms/cm²), after tensile test.

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13. ABSTRACT (Maximum 200 words) The International Space Station (ISS) solar arrays utilize MD-944 diode tape with silicone pressure-sensitive adhesive to protect the underlying diodes and also provide a high-emittance surface. Onorbit, the silicone adhesive will be exposed and ultimately convert to a glass-like silicate due to atomic oxygen (AO). The current operational plan is to retract ISS solar array P6 and leave it stored under load for a long duration (6 mo or more). The exposed silicone adhesive must not cause the solar array to stick to itself or cause the solar array to fail during redeployment. The Environmental Effects Branch at Marshall Space Flight Center, under direction from the ISS Program Office Environments Team, performed simulated space environment exposures with 5-eV AO, near ultraviolet radiation and ionizing radiation. The exposed diode tape samples were put under preload and then the resulting blocking force was measured using a tensile test machine. Test results indicate that high-energy AO, ultraviolet radiation, and electron ionizing radiation exposure all reduce the blocking force for a silicone-to-silicone bond. AO exposure produces the most significant reduction in blocking force.				
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